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SAFETY FIRST, LAST, AND ALWAYS

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The organic chemistry laboratory is potentially one of the most dangerous of undergraduate laboratories. That is why you must have a set of safety guidelines. It is a very good idea to pay close attention to these rules, for one very good reason:

The penalties are only too real.

Disobeying safety rules is not at all like flouting many other rules. *You can get seriously hurt.* No appeal. No bargaining for another 12 points so you can get into medical school. Perhaps as a patient, but certainly not as a student. So, go ahead. Ignore these guidelines. But remember—

You have been warned!

1. ***Wear your goggles.*** Eye injuries are extremely serious and can be mitigated or eliminated if you keep your goggles on *at all times*. And I mean *over your eyes*, not on top of your head or around your neck. There are several types of eye protection available, some of it acceptable, some not, according to local, state, and federal laws. I like the clear plastic goggles that leave an unbroken red line on your face when you remove them. Sure, they fog up a bit, but the protection is superb. Also, think about getting chemicals or chemical fumes trapped under your contact lenses before you wear them to lab. Then don't wear them to lab. Ever.
2. ***Touch not thyself.*** Not a Biblical injunction, but a bit of advice. You may have just gotten chemicals on your hands, in a concentration that is not noticeable, and sure enough, up go the goggles for an eye wipe with the fingers. Enough said.
3. ***There is no "away."*** Getting rid of chemicals is a very big problem. You throw them from here, and they wind up poisoning someone else. Now there are some laws to stop that from happening. The rules were really designed for industrial waste, where there are hundreds of gallons of waste that have the same composition. In a semester of organic lab there will be much smaller amounts of different materials. Waste containers could be provided for everything, but this is not practical. If you don't see the waste can you need, ask your instructor. When in doubt, *ask*.
4. ***Bring a friend.*** *You must never work alone.* If you have a serious accident and you are all by yourself, you might not be able to get help before you die. Don't work alone, and don't work at unauthorized times.

5. ***Don't fool around.*** Chemistry is serious business. Don't be careless or clown around in lab. You can hurt yourself or other people. You don't have to be somber about it; just serious.
6. ***Drive defensively.*** Work in the lab as if someone else were going to have an accident that might affect you. Keep the goggles on because *someone else* is going to point a loaded, boiling test tube at you. *Someone else* is going to spill hot, concentrated acid on your body. Get the idea?
7. ***Eating, drinking, or smoking in lab.*** Are you kidding? Eat in a chem lab? Drink in a chem lab??? Smoke, and blow yourself up????
8. ***Keep it clean.*** Work neatly. You don't have to make a fetish out of it, but try to be *neat*. Clean up spills. Turn off burners or water or electrical equipment when you're through with them.
9. ***Where it's at.*** Learn the location and proper use of the fire extinguishers, fire blankets, safety showers, and eyewash stations.
10. ***Making the best-dressed list.*** Keep yourself covered from the neck to the toes—no matter what the weather. That might include long-sleeved tops that also cover the midsection. Is that too uncomfortable for you? How about a chemical burn to accompany your belly-button, or an oddly shaped scar on your arm in lieu of a tatoo. And pants that come down to the shoes, covering any exposed ankles is probably a good idea as well. No open-toed shoes, sandals, or canvas-covered footwear. No loose-fitting cuffs on the pants or the shirts. Nor are dresses appropriate for lab, guys. Keep the midsection covered. Tie back that long hair. And a small investment in a lab coat can pay off, projecting that extra professional touch. It gives a lot of protection too. Consider wearing disposable gloves. Clear polyethylene ones are inexpensive, but the smooth plastic is slippery, and there's a tendency for the seams to rip open when you least expect it. Latex examination gloves keep the grip and don't have seams, but they cost more. Gloves are not perfect protectors. Reagents like bromine can get through and cause severe burns. They'll buy you some time though and can help mitigate or prevent severe burns. Oh, yes—laboratory aprons: they only cover the *front*, so your exposed legs are still at risk from behind.
11. ***Hot under the collar.*** Many times you'll be asked or told to heat something. Don't just automatically go for the Bunsen burner. That way lies *fire*. Usually—

No Flames!

Try a hot plate, try a heating mantle (see Chapter 18, "Sources of Heat"), but try to stay away from flames. Most of the fires I've had to put out started when some bozo decided to heat some flammable solvent in an open beaker. Sure, there are times when you'll **HAVE** to use a flame, but use it away from all flammables and in a hood (Fig. 1), and only with the permission of your instructor.

12. **Work in the hood.** A hood is a specially constructed workplace that has, at the least, a powered vent to suck noxious fumes outside. There's also a safety glass or plastic panel you slide down as protection from exploding apparatus (Fig. 1). If it is at all possible, treat every chemical (even solids) as if toxic or bad-smelling fumes came from it, and carry out as many of the operations in the organic lab as you can *inside a hood*, unless told otherwise.
13. **Keep your fingers to yourself.** Ever practiced "finger chemistry"? You're unprepared so you have a lab book out, and your finger points to the start of a sentence. You move your finger to the end of the first line and do that operation—

"Add this solution to the beaker containing the ice-water mixture"

And WHOOSH! Clouds of smoke. What happened? The next line reads—

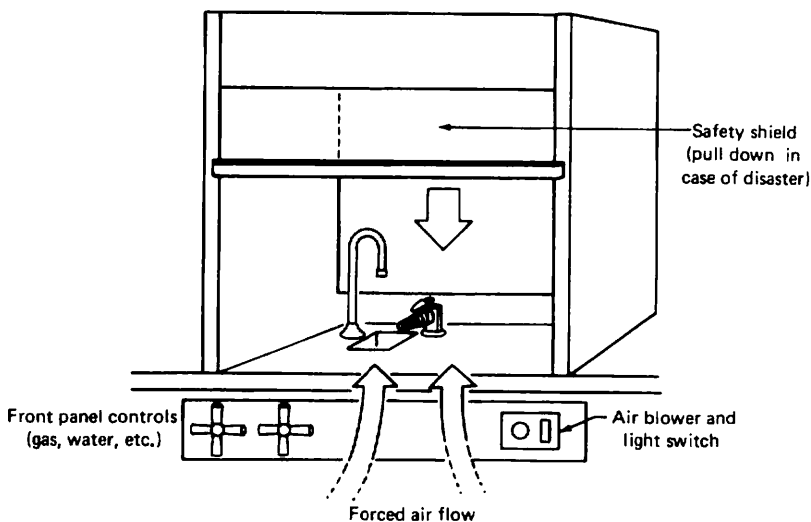


Fig. 1 A typical hood.

“very carefully as the reaction is highly exothermic”

But you didn't read that line, or the next, or the next. So you are a danger to yourself and everyone else. Read and take notes on any experiment before you come to lab (see Chapter 2, “Keeping a Notebook”).

14. ***What you don't know can hurt you.*** If you are not sure about an operation, or you have any question about handling anything, *please* ask your instructor before you go on. Get rid of the notion that asking questions will make you look foolish. Following this safety rule may be the most difficult of all. Grow up. Be responsible for yourself and your own education.
15. ***Blue Cross or Blue Shield?*** Find out how you would get medical help, if you needed it. Sometimes during a summer session, the school infirmary is closed and you would have to be transported to the nearest hospital.

These are a few of the safety guidelines for an organic chemistry laboratory. You may have others particular to your own situation.

ACCIDENTS WILL NOT HAPPEN

That's an attitude you might hold while working in the laboratory. You are **not** going to do anything, or get anything done to you, that will require medical attention. If you do get cut, and the cut is not serious, wash the area with water. If there's serious bleeding, apply direct pressure with a clean, preferably sterile, dressing. For a minor burn, let cold water run over the burned area. For chemical burns to the eyes or skin, flush the area with lots of water. In every case, get to a physician if at all possible.

If you have an accident, *tell your instructor immediately. Get help!* This is no time to worry about your grade in lab. If you put grades ahead of your personal safety, be sure to see a psychiatrist after the internist finishes.

DISPOSING OF WASTE

Once you do your reaction, since your mother probably doesn't take organic lab with you, you'll have to clean up after yourself. I've hesitated to write

this section for a very long time because the rules for cleaning up vary greatly according to, but not limited to, Federal, state, and local laws, as well as individual practices at individual colleges. There are even differences—legally—if you or your instructor do the cleaning up. And, as always, things do seem to run to money—the more money you have to spend, the more you can throw away. So there's not much point to even trying to be authoritative about waste disposal in this little manual, but there are a few things I have picked up that you should pay attention to. Remember, my classification scheme may not be the same as the one you'll be using. When in doubt, **ask! Don't just throw everything into the sink. Think.**

Note to the picky: The word *nonhazardous*, as applied here, means relatively benign, as far as organic laboratory chemicals go. After all, even pure water, carelessly handled, can kill you.

1. ***Nonhazardous insoluble waste.*** Paper, corks, sand, alumina, silica gel, sodium sulfate, magnesium sulfate, and so on can probably go into the ordinary wastebaskets in the lab. Unfortunately, these things can be contaminated with hazardous waste (see discussion below), and then they need special handling.
2. ***Nonhazardous soluble solid waste.*** Some organics, such as benzoic acid, are relatively benign and can be dissolved with a lot of tap water and flushed down the drains. But if the solid is that benign, it might just as well go out with the nonhazardous insoluble solid waste, no? Check with your instructor; watch out for contamination with more hazardous materials.
3. ***Nonhazardous soluble liquid waste.*** Plain water, down the drains, as well as water-soluble substances not otherwise covered below. Ethanol can probably be sent down the drains, but butanol? It's not that water soluble, so it probably should go into the general organic waste container. Check with your instructor; watch out for contamination with more hazardous materials.
4. ***Nonhazardous insoluble liquid waste.*** Compound such as 1-butanol (discussed above), diethyl ether, and most other solvents and compounds not covered otherwise. In short, this is the traditional "organic waste" category.
5. ***Generic hazardous waste.*** Pretty much all else not listed separately. Hydrocarbon solvents (hexane, toluene), amines (aniline, triethylamine), amides, esters, acid chlorides, and on and on. Again,

traditional “organic waste.” Watch out for incompatibilities, though, before you throw just anything in any waste bucket. If the first substance in the waste bucket was acetyl chloride and the second was diethylamine (both hazardous liquid wastes), the reaction may be quite spectacular. You may have to use separate hazardous waste containers for these special circumstances.

6. **Halogenated organic compounds.** 1-Bromobutane and t-butyl chloride, undergraduate laboratory favorites, should go into their own waste container as “halogenated hydrocarbons.” There’s a lot of agreement on this procedure for these simple compounds. But what about your organic unknown, 4-bromobenzoic acid? I’d have you put it, and any other organic with a halogen, in the “halogenated hydrocarbon” container and not flush it down the drain as a harmless organic acid, as you might do with benzoic acid.
7. **Strong inorganic acids and bases.** Neutralize them, dilute them, and flush them down the sink. At least as of this writing.
8. **Oxidizing and reducing agents.** Reduce the oxidants and oxidize the reductants before disposal. Be careful! Such reactions can be highly exothermic. Check with your instructor before proceeding.
9. **Toxic heavy metals.** Convert to a more benign form, minimize the bulk, and put in a separate container. If you do a chromic acid oxidation, you might reduce the more hazardous Cr^{+6} to Cr^{+3} in solution and then precipitate the Cr^{+3} as the hydroxide, making lots of expensive-to-dispose-of chromium solution into a tiny amount of solid precipitate. There are some gray areas, though. Solid manganese dioxide waste from a permanganate oxidation should probably be considered a hazardous waste. It can be converted to a soluble Mn^{+2} form, but should Mn^{+2} go down the sewer system? I don’t know the effect of Mn^{+2} (if any) in the environment. But do we want it out there?

Mixed Waste

Mixed waste has its own special problems and raises even more questions. Here are some examples:

1. **Preparation of acetaminophen (Tylenol): A multi-step synthesis.** You’ve just recrystallized 4-nitroaniline on the way to acetaminophen, and washed and collected the product on your Buchner funnel. So you

have about 30–40 mL of this really orange solution of 4-nitroaniline and byproducts. The nitroaniline is very highly colored, the byproducts probably more so, so there isn't really a lot of solid organic waste in this solution; not more than perhaps a hundred milligrams or so. Does this go down the sink, or is it treated as organic waste? Remember, you have to package, label, and transport to a secure disposal facility what amounts to 99.9% perfectly safe water. Check with your instructor.

2. ***Preparation of 1-bromobutane.*** You've just finished the experiment and you're going to clean out your distillation apparatus. There is a residue of 1-bromobutane coating the three-way adapter, thermometer, inside of the condenser, and the adapter at the end. Do you wash the equipment in the sink and let this minuscule amount of a halogenated hydrocarbon go down the drain? Or do you rinse everything with a little acetone into yet another beaker and pour that residue into the "halogenated hydrocarbon" bucket, fully aware that most of the liquid is acetone and doesn't need special halide treatment? Check with your instructor.
3. ***The isolation and purification of caffeine.*** You've dried a methylene chloride extract of caffeine and are left with methylene chloride saturated drying agent. Normally a nonhazardous solid waste, no? Yes. But where do you put it while the methylene chloride is on it? Some would have you put it in a bucket in a hood and let the methylene chloride evaporate into the atmosphere. Then the drying agent is nonhazardous solid waste. But you've merely transferred the problem somewhere else. Why not just put the whole mess in with the "halogenated hydrocarbons"? Usually "halogenated hydrocarbons" go to a special incinerator equipped with traps to remove HCl or HBr produced upon burning. Drying agents don't burn very well, and the cost of shipping the drying agent part of this waste is very high.

Question: Is this cost higher than that of letting the methylene chloride evaporate into the atmosphere? The current and past Republican administrations see fit to keep studying the interactions of pollutants in the atmosphere rather than actually do anything that might disturb "business as usual," so this method of pollution control is, at least, highly patriotic. What should you do? Again, ask your instructor.

In these cases, as in many other questionable situations, I tend to err on the side of caution and consider the bulk of the waste to have the attributes of its most hazardous component. This is, unfortunately, the most expensive way to look at the matter. In the absence of guidelines:

1. Don't make a lot of waste in the first place.
2. Make it as benign as possible. (Remember though, such reactions can be highly exothermic, so proceed with caution.)
3. Reduce the volume as much as possible.

Oh. Try to remember that sink drains can be tied together, and if you pour a sodium sulfide solution down one sink, while someone else is diluting an acid in another sink, toxic, gagging, rotten-egg-smelling hydrogen sulfide can back up the drains in your entire lab, and maybe even the building.